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PATENT SPECIFICATION

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DRAWINGS ATTACHED

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(54) CONTINUOUS PROCESS FOR THE PRODUCTION OF SACCHAROSE ESTERS OF FATTY ACIDS

(71) I. ROSHDY ISMAIL, of Rotterstrasse 6, Spich, West Germany, a German Citizen, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:-

This invention relates to a continuous process for the production of saccharose esters of fatty acids.

Published German Patent Specification No. P 15 18 251.-1-42 described a process for the production of saccharose fatty acid esters, in which the saccharose is subjected to ester interchange with alkyl esters of fatty acids, which contain 12-22 carbon atoms, in a liquid two-phase system, where the one phase consists at the beginning of the reaction of the solution of the fatty acid alkyl ester in an aliphatic or aromatic hydrocarbon, and the other phase contains the saccharose and possibly the catalyst in solution in a polar solvent. Dimethyl sulphoxide or dimethyl formamide are preferred as polar solvents. Moreover the mixture ratio of liquid hydrocarbon to polar solvent should be between 0.5 : 1 and 5 : 1 and especially 2 : 1 and 2.5 : 1. Discontinuous process method are described in the examples of execution of this patent application.

It has been found that one can advantageously carry out the process continuously. Accordingly, the invention provides a process for the continuous production of a saccharose fatty acid ester in which:

(1) a solution of saccharose with 0.05 to 2% by weight of an alkaline catalyst in a polar solvent, together, with (2) a solution, prepared separately, of a fatty acid ester, derived from a C₁₂₋₂₂ straight chain saturated or unsaturated fatty acid and a C₁₋₈ aliphatic or cycloaliphatic, mono- or poly-hydric alcohol, in an aliphatic or aromatic hydrocarbon or chloro-hydrocarbon having a boiling point between 70 and 250°C, are fed continuously into a series of at least three reaction zones connected in cascade, in which the reagents (1) and (2) are intensively intermixed at a temperature between 40 and

150°C, any alcohol liberated being distilled off under reduced pressure; the mixture issuing from the last reaction zone after a mean residence time of between 1 and 15 hours is passed to a settling vessel where it divides into two phases; the polar solvent phase, containing the saccharose ester, is removed and concentrated by evaporation under reduced pressure in a drier; and the polar solvent which is in the drier is fed back for re-use earlier in the process. The solvents recovered from the settling vessel or evaporating device can be returned to the process without intermediate purification.

Polar solvents which can be used are N-methyl-morpholine, triethyl amine, pyridine, quinoline, pyrazine, N-methyl pyrazine, N,N'-dimethyl-piperazine, 2-pyrrolidone, N-methyl-pyrrolidone, N,N'-dimethyl-formamide, and dimethyl sulphoxide, which is used for preference.

By way of example, toluene, ethylene, ethyl benzene, cyclohexyl-benzene, nonyl benzene, benzine and ligroin are suitable as aromatic and aliphatic hydrocarbons which boil between 100 and 200°C. By way of example, dichloroethane, perchloro-ethylene, tetrachloro-ethane and dichloro-benzene come under consideration as chloro-hydrocarbons. The weight ratio of polar solvent to hydrocarbon or chloro-hydrocarbon expediently amounts to between 0.5 : 1 and 5 : 1, especially 1.5 : 1 and 2.5 : 1. Hydrocarbons are preferably used as non-polar solvents.

Advantageously fatty acid esters in which the fatty acid comprises a weakly hydrophilic group, as for example a hydroxyl, carbonyl, oxime or alkoxy group are used. The methyl, ethyl, propyl, isopropyl or cyclohexyl esters of 12-hydroxy-stearic acid or of ricinoleic acid are examples of such fatty acid esters. In place of the esters of mono-valent alcohols it is also possible to use esters derived from multi-valent alcohols, especially fatty acid triglycerides.

Substances having an alkaline reaction, especially potassium carbonate or potassium or sodium alcoholates, in quantities between

0.05 and 2% by weight of the quantity of fatty acid ester, are added as catalysts for the ester interchange.

The dissolving of the saccharose in the polar solvent is more expediently effected at elevated temperature, for example 70-90°C. The quantity regulation of the two solutions is such that the molar ratio of saccharose to fatty acid ester preferably amounts to 1 : 1. The reaction in the reaction cascade is carried out at temperatures between 40 and 150°, preferably between 80 and 90°C. The low alcohol liberated during the interchange distills off and is received in a cooling device. The throughflow through the reaction cascade is regulated so that a mean residence time of 1-1.5 hours, preferably 3-10 hours, is produced. During the reaction, the reagents should be constantly intensively mixed, in order to achieve the most rapid and complete reaction possible.

If triglycerides are used as starting materials, then in the reaction mixture the equimolar quantity of diglyceride forms as well as the saccharose ester. The diglyceride is substantially dissolved in the hydrocarbon phase and can be recovered therefrom in the subsequent working up of the solvent.

The mixture issuing from the reaction cascade divides itself up relatively quickly into a lighter phase consisting of the hydrocarbon and a heavier phase containing the polar solvent and the saccharose ester. The hydrocarbon can be returned to the process as solvent for the fatty acid ester. The concentration by evaporation of the solution containing the saccharose ester is effected continuously, while ordinary driers provided with mixing or atomising elements can be used, like spray, sprinkle and cascade driers or rotating drum driers or drum driers equipped with scraper devices. The temperature during the drying should not substantially exceed 80 to 100°C., in order to avoid discoloration of the sugar ester. The polar solvent distilled-off is likewise returned to the process.

The invention will be further explained hereinafter with reference to the flow diagram which is illustrated in the accompanying drawing.

In the drawing, the sugar and the alkaline catalyst are dissolved in the polar solvent in the heatable agitating vessel 1 and are fed by way of the quantity-regulating pump 4 into the reaction cascade 5. The dissolving of the fatty acid ester in the hydrocarbon takes place in the agitating vessel 2, whereupon the solution is transferred by way of the quantity regulating pump 3 likewise into the reaction cascade. The cascade 5 consists of several heatable containers equipped with an agitating mechanism, the overflow of each vessel being connected to the subsequent reaction

container. The low alcohol liberated in the transesterification is cooled and recovered in the condenser 9. The mixture issuing from the last reaction vessel travels into the settling vessel 6, whence the hydrocarbon collected beyond an overflow is returned by way of a quantity-regulating pump 15 into the dissolving vessel 2. The solution containing the sugar ester is evaporated for drying in the thin-layer evaporator 7, and the ester obtained is cooled on a cooling roll 8. The solvent condensed in the condenser 11 and collected in the receiver 12 is likewise returned by way of the quantity-regulating pump 14 into the dissolving vessel 1.

Example 1

The experimental apparatus incorporated five heatable vessels provided with intensive agitators and connected one after the other, each having a filling capacity to the overflow pipe of 2 litres. A mixture consisting of a solution of 165 g. of saccharose and 3 g. of potassium carbonate in 500 g. of dimethyl sulphoxide and a solution of 152 g. of 12-hydroxy - stearic methyl ester in 300 g. of benzene (boiling point range : 100 to 140°C.) were fed hourly into the first vessel. A temperature of 85°C. and a pressure of 100 Torr were maintained in the apparatus. The mean residence time of the mixture to be subjected to ester interchange in the apparatus amounted to 8.9 hours.

The mixture flowing out of the last-agitating vessel was transferred into a settling vessel. This consisted of a vertically arranged cylinder which was equipped with a centrally fitted supply conduit and a lower outlet cock for the dimethyl sulphoxide solution and an overflow pipe for the hydrocarbon. The overflow pipe and the outlet cock led to two spacious collecting containers, from which the solutions were withdrawn from time to time. The dimethyl sulphoxide was distilled off *in vacuo* and the saccharose ester remaining behind was dried at a pressure of 0.1 Torr. The yield of saccharose ester was practically quantitative, since no more fatty acid ester was contained in the benzene phase.

The sugar ester obtained melted at 56 to 60°C. and possessed the following analysis data:

surface tension: 37 dyn/cm. at 30°C. in 0.15% solution
saponification number: 90
acid number: 0

Example 2

1 litre of a mixture consisting of 1 part by weight of a 55.5% by weight solution of castor oil in benzene (boiling point range 100 to 140°C.) and 1.63 parts by weight of a 12.5% by weight solution of saccharose in dimethyl sulphoxide, which also contained 0.08% by weight of potassium carbonate, was

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10	75	15	15
15			
20	85	20	20
25	90	25	25
30			
35	95	30	30
40	100		
45			
50	105		
55			
60	110		
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	120		
	125		

fed hourly into the apparatus as described in Example 1. The temperature in the apparatus was 85°C., the pressure 1 atm., and the mean residence time of the mixture 10 hours.

The mixture issuing from the last vessel was separated and worked up as described in Example 1. The sugar ester, obtained with 96% yield, possessed a surface tension of 40 dyn/cm. at 30°C. in 0.15% solution. The equivalent quantity of ricinoleic fatty acid diglyceride was isolated out of the benzine phase.

The sugar esters obtained by these processes are of a kind suitable for use in the detergent and perfume fields, for reducing surface tension.

WHAT I CLAIM IS:—

1. A process for the continuous production of a saccharose fatty acid ester in which: (1) a solution of saccharose with 0.05 to 2% by weight of an alkaline catalyst in a polar solvent, together with (2) a solution, prepared separately, of a fatty acid ester, derived from a C₁₂₋₁₂ straight chain saturated or unsaturated fatty acid and a C₁₋₆ aliphatic or cycloaliphatic, mono- or poly-hydric alcohol, in an aliphatic or aromatic hydrocarbon or chloro-hydrocarbon having a boiling point between 70 and 250°C., are fed continuously into a series of at least three reaction zones connected in cascade, in which the reagents (1) and (2) are intensively intermixed at a temperature between 40 and

150°C. any alcohol liberated being distilled off under reduced pressure; the mixture issuing from the last reaction zone after a mean residence time of between 1 and 15 hours is passed to a settling vessel where it divides into two phases; the polar solvent phase, containing the saccharose ester, is removed and concentrated by evaporation under reduced pressure in a drier; and the polar solvent which is liberated in the drier is fed back for re-use earlier in the process.

2. A process according to Claim 1, characterised in that the mean residence time is 3-10 hours in the reaction zones.

3. A process according to Claim 1 and 2, characterised in that dimethyl sulphoxide is used as polar solvent and a hydrocarbon is used as non-polar solvent.

4. A process according to any of Claims 1 to 3, wherein the flow of the two solutions is adjusted so that the molar ratio of saccharose to fatty acid ester is 1:1.

5. A process according to any of Claims 1 to 4, wherein said temperature is 80-90°C.

6. A process for the production of saccharose fatty acid esters substantially as herein described with reference to the accompanying drawings.

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COMPLETE SPECIFICATION

1 SHEET

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